

### Observation:

I] Standardisation of 0.1 N HCl solutions:

- i) Solution in conical flask : 25 cm<sup>3</sup> diluted solution 0.05 N borax solution + 2 drops of 1% methyl orange indicator + 15 cm<sup>3</sup> of D.W.  
 ii) Solution in Burette : 0.1 N HCl solution.  
 iii) Indicator : 1% methyl orange indicator.  
 iv) End point : Yellow to pink colour.

### Observation Table:

Level	Burette Reading (cm <sup>3</sup> )			C.B.R. (cm <sup>3</sup> )
	I	II	III	
Initial	0.0	0.0	0.0	8.0 cm <sup>3</sup>
Final	8.0	8.1	8.0	
Difference	8.0	8.1	8.0	

II] Estimation of Na<sub>2</sub>CO<sub>3</sub> in washing soda:

- i) Solution in burette : 0.1 N HCl solution.  
 ii) Solution in beaker : 10 cm<sup>3</sup> of diluted solution of washing soda + 25 cm<sup>3</sup> of distilled water.

Obs. No.	Volume of 0.1 N HCl solution added in cm <sup>3</sup>	Volume (V <sub>1</sub> ) change (V <sub>2</sub> - V <sub>1</sub> ) cm <sup>3</sup>	Corrected volume V <sub>cor</sub> = V + ΔV <sub>cor</sub>	pH	Δ pH	$\frac{\Delta pH}{\Delta V}$
1	0.0	-		7.87	-	-
2	0.5	0.5	1	7.54	0.33	0.66
3	1.0	0.5	1.5	7.34	0.2	0.4
4	1.5	0.5	2	7.00	0.34	0.68
5	2.0	0.5	2.5	6.39	0.61	1.22
6	2.5	0.5	3	6.18	0.21	0.42
7	3.0	0.5	3.5	6.15	0.03	0.06
8	3.5	0.5	4	5.65	0.5	1
9	4.0	0.5	4.5	5.38	0.27	0.54
10	4.5	0.5	5	4.16	1.22	2.44
11	5.0	0.5	5.5	3.98	0.18	0.36

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Aim: To determine percentage purity of sodium carbonate in washing soda. pH metrically.

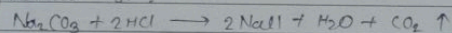
### Requirements:

Washing soda powder, 0.05 N borax solution, 0.1 N HCl solution, 1% methyl orange indicator, buffer sol<sup>n</sup> of pH 4.01 and 9.18, pH meter with electrode assembly, burette, 10 cm<sup>3</sup> and 25 cm<sup>3</sup> pipette, standard measuring flask, 100 cm<sup>3</sup> beaker conical flask etc.

### Theory:

Na<sub>2</sub>CO<sub>3</sub> is salt of weak acid (Carbonic acid) and strong base (NaOH). As a result, in aqueous solution, it hydrolysis to form a basic solution.

The strength and amount of Na<sub>2</sub>CO<sub>3</sub> can be estimated with fair accuracy using pH metric titration technique against a strong acid like HCl. The reaction can be represented as:



During the course of titration, pH is noted and a graph of pH against the volume of titrant added is plotted and from it equivalence point is determined graphically.

for accurate determination of equivalence point, a first derivatives graph of pH vs V.

### Procedure:

A] i) Preparation of sample solution:

i) Weigh accurately 0.5 g of (W<sub>1</sub>) of the given sample of



12	5.5	0.5	6	3.85	0.18	0.24
13	6.0	0.5	6.5	2.97	0.13	1.76
14	6.5	0.5	7	2.89	0.88	0.16
15	7.0	0.5	7.5	2.85	0.08	0.08
16	7.5	0.5	8	2.82	0.04	0.06
17	8.0	0.5	8.5	2.78	0.03	0.08
18	8.5	0.5	9	2.72	0.04	0.12
19	9.0	0.5	9.5	2.69	0.06	0.12
20	9.5	0.5	10	2.98	0.31	0.62
21	10.0	0.5	10.5	2.24	0.14	0.28
22	10.5	0.5	11	2.20	0.04	0.08

∴ Volume of HCl solution req. for complete titration ( $V_2$ ) = 2.5 cm<sup>3</sup>

Calculation:-

Amount of  $\text{Na}_2\text{CO}_3$  in the given washing soda sample 2 molar of HCl are req. neutralise 1 mole of  $\text{Na}_2\text{CO}_3$

∴ 2000 cm<sup>3</sup> of 1 N HCl sol<sup>n</sup> = 106 g of  $\text{Na}_2\text{CO}_3$

$$2.5 \text{ of } 0.15 \text{ N sol}^n = \frac{106 \times V_2 \times A}{2000}$$

$$= \frac{106 \times 8.5 \times 0.15}{2000}$$

$$= 0.067$$

$$\therefore y = 0.067 \text{ of } \text{Na}_2\text{CO}_3$$

10 cm<sup>3</sup> of diluted sample sol<sup>n</sup> contains 0.067 mg of  $\text{Na}_2\text{CO}_3$

$$\therefore 100 \text{ cm}^3 = 0.067 \times 10 \text{ mg of } \text{Na}_2\text{CO}_3$$

$$= 0.67 \text{ of } \text{Na}_2\text{CO}_3$$

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washing soda & transfer it to a 100 cm<sup>3</sup> beaker

2) Dissolve it in minimum quantity of distilled water. Transfer the solution to 100 cm<sup>3</sup> standard measuring flask dilute it to the mark with distilled water.

ii) Preparation of 0.05 N borax solution:

1) Weigh accurately 0.381 g of borax and transfer it to a clean dry 100 cm<sup>3</sup> beaker.

2) Dissolve it in a minimum quantity of distilled water, transfer it 100 cm<sup>3</sup> standard measuring flask and dilute it upto the mark with distilled water.

B) Standardization of 0.1 N HCl solution:-

3) Rinse and fill a burette with the supplied 0.1 N HCl solution.

4) Pipette out 25 cm<sup>3</sup> of 0.05 N borax solution in a clean dry 150 cm<sup>3</sup> conical flask. Add 2 drops 1% methyl orange indicator and about 10-15 cm<sup>3</sup> solution from the burette. End point will be from yellow to pink colour. (x cm<sup>3</sup>)

C) Estimation of  $\text{Na}_2\text{CO}_3$  in washing soda sample.

7) Pipette out 10 cm<sup>3</sup> of washing soda sample solution in a 100 cm<sup>3</sup> beaker. Add 25 cm<sup>3</sup> of distilled water to it.

8) Standardize the pH meter at pH 9.18 and 4.10 Using buffer solution of borax and potassium hydrogen phthalate respectively.

9) Wash and dry the electrodes by using them with

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A] Normality of HCl solution:

$$N_1 V_1 = N_2 V_2$$

$$N_1 \times 8.0 = 0.05 \times 2.5$$

$$N_1 = \frac{0.05 \times 2.5}{8.0}$$

$$N_1 = 0.15 \text{ N}$$

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tissue paper and dip in sample solution. stir the solution and record initial pH of the solution

10) Add 1.0 cm<sup>3</sup> of 0.1 N HCl solution till two distinct sharp pH are observed.

11) Add HCl solution dropwise and continuously stir the solution.

12) plot graphs of i) pH against volume of HCl solution added in cm<sup>3</sup> (ii)  $\Delta \text{pH}$  against mean volume of titrant added.

13) Determine the volume of  $V_1$  and  $V_2$  from the graph corresponding to half and complete neutralisation.

14) from the volume of  $V_2$  calculate the amount of  $\text{Na}_2\text{CO}_3$  present in washing soda sample.



Results:

1) 10 cm<sup>3</sup> of the diluted sample sol<sup>n</sup> required  
= 8.5 cm<sup>3</sup> of 0.1 N HCl solution.

2) Amount of Na<sub>2</sub>CO<sub>3</sub> in washing soda sample 0.67 mg  
of Na<sub>2</sub>CO<sub>3</sub> sol<sup>n</sup> in 100 cm<sup>3</sup> sample.

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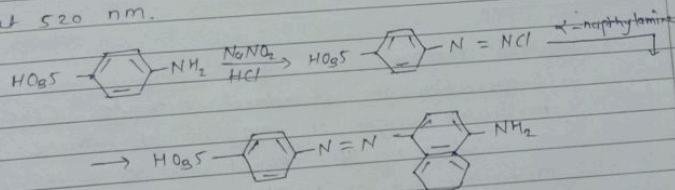
Aim:

To determine the amount of nitrite present in the given water sample colorimetrically.

Theory:

The presence of nitrite ions ( $\text{NO}_2^-$ ) in water sample may cause serious problems. It may react with dissolve oxygen affecting aquatic life. Therefore a concentration of  $\text{NO}_2^-$  ion in water should be brought to a reasonable limit. WHO has suggested that the upper limit for it in drinking water has 80 ppm.

When a dilute acid solution of sulphanilic acid and  $\alpha$ -naphthylamine are added upon by  $\text{NO}_2^-$  ion or  $\text{HNO}_2$  a red colour is developed which has absorption maximum at 520 nm.



The sulphanilic acid is converted into the corresponding diazo compounds, which couples with  $\alpha$ -naphthylamine to form a red coloured azo dye. So the intensity of colour developed (absorbance) is directly proportional to the concentration of nitrite nitrogen ion in water, so it, can be used to determine the amount of nitrite present in the water sample.

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Observation Table:

Sl. No.	Volume of				Final Volume with D.W. in cm <sup>3</sup>	Conc. of nitrate ions in Ppm.	Absorbance (A)
	10 ppm of nitrite solution in cm <sup>3</sup>	Sulphuric acid solution in cm <sup>3</sup>	$\alpha$ -naphthyl amine solution in cm <sup>3</sup>	2M sodium acetate solution in cm <sup>3</sup>			
1	0	2	2	2	100	0.0	0.04
2	1	2	2	2	100	0.1	0.34
3	2	2	2	2	100	0.2	0.40
4	3	2	2	2	100	0.3	0.52
5	4	2	2	2	100	0.4	0.56
6	5	2	2	2	100	0.5	0.62
7	10 ppm of Unknown	2	2	2	100	$x=0.18$	$As=0.23$

### Calculation:

#### 1) Solution Prepare:

Preparation of standard nitrate (1000 ppm)  
 molecular weight of  $\text{mg/cm}^3$  of nitrate  
 $\therefore 1000 \text{ mg} / 1000 \text{ cm}^3$  of nitrate = 1000 ppm.  
 Molecular wt. of  $\text{NaNO}_2 = 68.99$   
 wt. of nitrate = 46

$$\text{Weight (W)} = \frac{\text{molecular wt.} \times \text{conc. in ppm}}{\text{Atomic wt.} \times \text{no. of atom.}}$$

$$= \frac{68.99 \times 1000}{46 \times 1}$$

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### Requirements:

Sodium nitrite (A.R.) sulphuric acid,  $\alpha$ -naphthyl amine, sodium acetate HCl, pipette 1000 cm<sup>3</sup> and 100 cm<sup>3</sup> standard measuring flask, spectrophotometer, Cuvettes etc.

### Procedure:

#### A) Preparation of standard sol<sup>n</sup> and reagents:

- 1) Standard nitrite solution: Dissolve 4.98 g. of A.R. sodium nitrite in a minimum amount of distilled water. after shaking make-up to 1 dm<sup>3</sup>. This gives stock solution of nitrite ion. Dilute 10 cm<sup>3</sup> of this solution to 1000 cm<sup>3</sup> with distilled water in another standard measuring flask. This forms 10 ppm of standard nitrite nitrogen present.
- 2) Sulphanilic acid reagent: Dissolve 0.60 g of A.R. Sulphanilic acid in 100 cm<sup>3</sup> of 20% hydrochloric acid (v/v) in a 100 cm<sup>3</sup> standard measuring flask.
- 3)  $\alpha$ -naphthyl amine reagent: Dissolve 0.48 of A.R.  $\alpha$ -naphthyl amine in 100 cm<sup>3</sup> of 10% HCl (v/v) in a 100 cm<sup>3</sup> standard measuring flask.
- 4) 2M sodium acetate sol<sup>n</sup>: Dissolve 16.4 g of A.R. anhydrous sodium acetate in about 60 cm<sup>3</sup> of distilled water and make up the volume to 100 cm<sup>3</sup> using 100 cm<sup>3</sup> standard measuring flask (Always use fresh distilled water).

#### B) Preparation of Calibration Curve:

- 1) Take 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 cm<sup>3</sup> of 10 ppm standard nitrite nitrogen sol<sup>n</sup> in six serially numbered 100 cm<sup>3</sup> standard measuring flask.

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$$\text{Weight (W)} = \frac{68.99 \times 1000}{46}$$

$$= 1499.78 \text{ mg/2000 Lit.}$$

$$= 1.4997 \text{ gm/1000 Lit.}$$

$$\text{Weight} = \underline{0.374 \text{ gm/100 ml}}$$

Calculation from graph :-

conc. of nitrate in water 0.18 ppm.

10 cm<sup>3</sup> of water sample contains 0.18 ppm.

∴ 100 cm<sup>3</sup> of water sample contains.

$$= 100 \times (\text{PPM})$$

$$= 100 \times 0.18$$

$$= \underline{18.0 \text{ mg/100 ml of nitrate.}}$$

Conc of nitrate nitrogen of 100 cm<sup>3</sup> of the diluted water sample sol<sup>n</sup> x = 0.18 ppm. i.e. 0.18 mg/1000 cm<sup>3</sup>

∴ 100 cm<sup>3</sup> of the diluted water sample sol<sup>n</sup> contains 0 x mg nitrogen.

Now, 14 mg of Nitrogen = 46 mg of nitrate.

$$\therefore 0. x \text{ mg of nitrogen} = \frac{46 \times 0.18}{14}$$

$$= 0.328 \times x$$

$$= 0.328 \times 0.18$$

$$= \underline{0.059 \text{ mg of nitrite (NO}_2\text{)}}$$

Concentration of Nitrate in the supplied water sample

$$\text{sol}^n = 328 \times 0.18 = \underline{59.04 \text{ mg.}}$$

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7) Add 1 cm<sup>3</sup> of sulphonic acid sol<sup>n</sup> to each flask. shake well and wait for 5 min.

8) Now to each flask, add 1 cm<sup>3</sup> of α-naphthyl amine solution followed by addition of 1 cm<sup>3</sup> of 2M sodium acetate solution. Wait for 2-3 min.

9) Dilute each solution with fresh distilled water upto the mark and shake well.

10) Switch on the spectrometer. set the wavelength at 520 nm.

11) Measure the absorbance (A) of each sol<sup>n</sup> against reagent blank prepared in step No. 5.

c) Analysis of sample solution:

1) Pipette out 1 cm<sup>3</sup> supplied water sample in another 100 cm<sup>3</sup> standard measuring flask.

2) Add 1 cm<sup>3</sup> of sulphonic acid, 1 cm<sup>3</sup> of α-naphthyl amine solution followed by 1 cm<sup>3</sup> of 2M sodium acetate sol<sup>n</sup> in the same manner as mentioned in step Nos. 2 & 3 above.

3) Dilute the solution with fresh distilled water upto the mark. shake the solution well wait for 5 min.

4) Determine the absorbance of water sample solution against reagent blank at 520 nm. Record the reading (As)

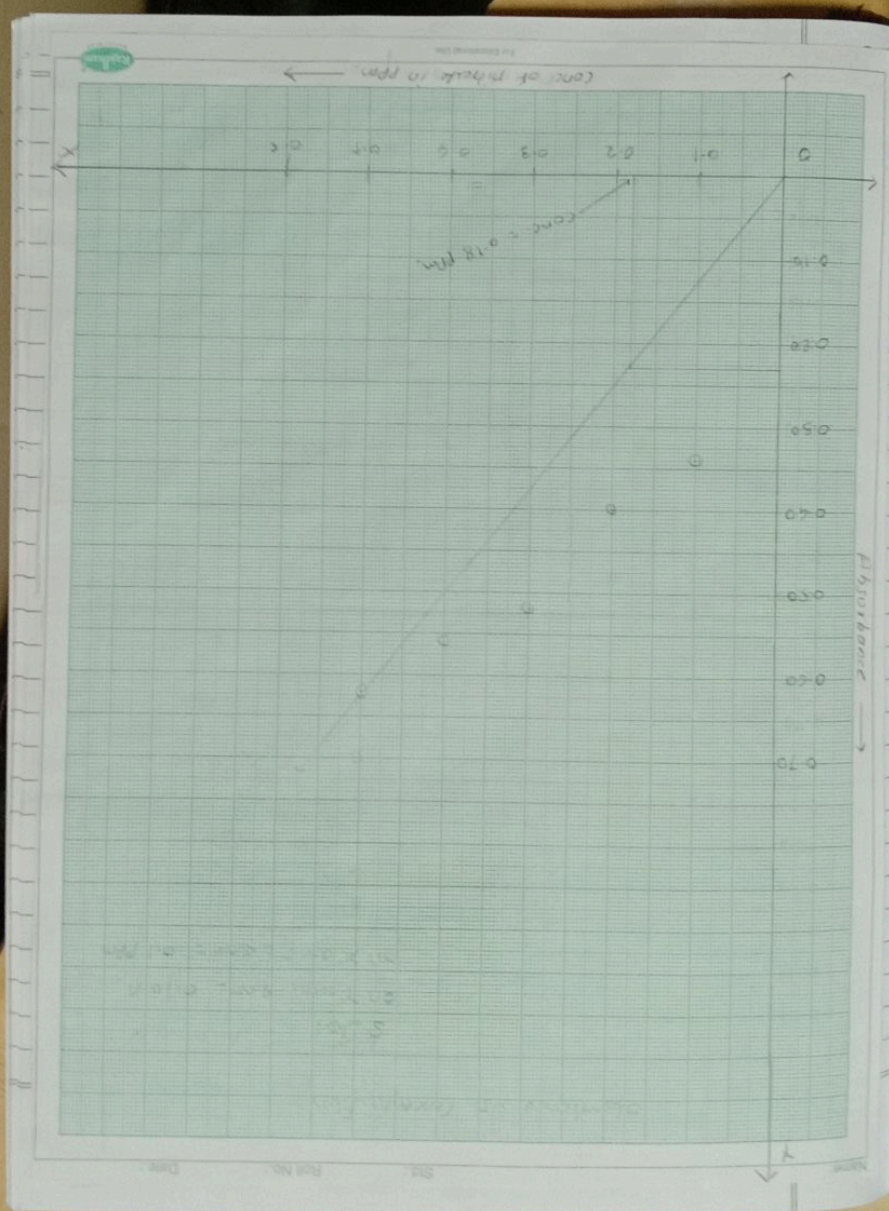
5) Plot a graph of absorbance of sample values (A) against concentration of nitrite nitrogen in ppm.

6) With the help of absorbance of sample solution measured (As) and calibration curve constructed, determine the amount of nitrite present in supplied water sample solution.

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- Result:
- The given water sample contain  $0.18 \text{ ppm}$  or  $18.0 \text{ mg/lit.}$
- 1) Absorbance of  $100 \text{ cm}^3$  of the diluted water sample solution (As) =  $0.23 A$
  - 2) Amount of Nitrite in  $1 \text{ cm}^3$  of water sample solution =  $0.059 \text{ mg.}$
  - 3) Concentration of Nitrite in the supplied water sample solution =  $59.04 \text{ mg.}$



Aim: Simultaneous determination of Fe (II) and Fe (III) by 1,10 phenanthroline method.

Requirements:

1) Standard Fe (II) solution.

Dissolve 0.176 g of (AR) ferrous ammonium sulphate in distilled water containing 2.5 cm<sup>3</sup> of conc. HCl Dilute the solution with distilled water to 250 cm<sup>3</sup>. This gives standard Fe (II) solution of strength 100 ppm.

2) 0.05% solution of 1,10 phenanthroline solution in D.L. 3) 10% Hydroxylamine hydrochloride soln.

4) Dissolve 1.021 g. of (AR) potassium hydrogen phthalate in 100 cm<sup>3</sup> of distilled to prepare 0.05M potassium hydrogen phthalate soln.

5) Sample solution containing a mixture of Fe (II) and Fe (III) in 100 cm<sup>3</sup> standard measuring flask.

6) 100 cm<sup>3</sup> standard measuring flasks, distilled water, barbituric acid pipettes, spectrophotometer 515 nm filter etc.

Theory:

Iron (II) reacts with 1,10 phenanthroline to form an orange-red coloured complex  $[\text{C}_{12}\text{H}_8\text{N}_6\text{Fe}]^{2+}$ . The colour intensity is dependent of the acidity in the pH range 2-9 and is stable for long periods. It shows absorption at 515 nm at which iron (III) has no absorption.

Both Fe (II) and Fe (III) ions can be determined at 515 nm with pH of about 4 by reducing Fe (III) to Fe (II) which will give absorption due to both Fe (II)



Graph: Total amount of iron in  $[Fe(II) + Fe(III)]$  ppm

$$x = \text{Total amount of iron} = 0.465 \text{ ppm}$$

$$y = \text{Amount of } Fe(II) \text{ in ppm} = 0.165 \text{ ppm}$$

$$z = (x - y) \text{ amount of } Fe(III) \text{ in ppm} = (0.465 - 0.165) = 0.3 \text{ ppm}$$

Calculation:  
 1) Amount of  $Fe(II)$  present in the 10 cm<sup>3</sup> of the diluted sample soln = 0.465 ppm.  
 $\therefore$  Amount of  $Fe(II)$  present in the given sample soln =  $10 \times 0.465 = 4.65 \text{ mg/100 cm}^3$   
 $\therefore$  100 cm<sup>3</sup> contains  $x \text{ mg} = 0.465 \text{ mg of } Fe(II) \text{ ions.}$

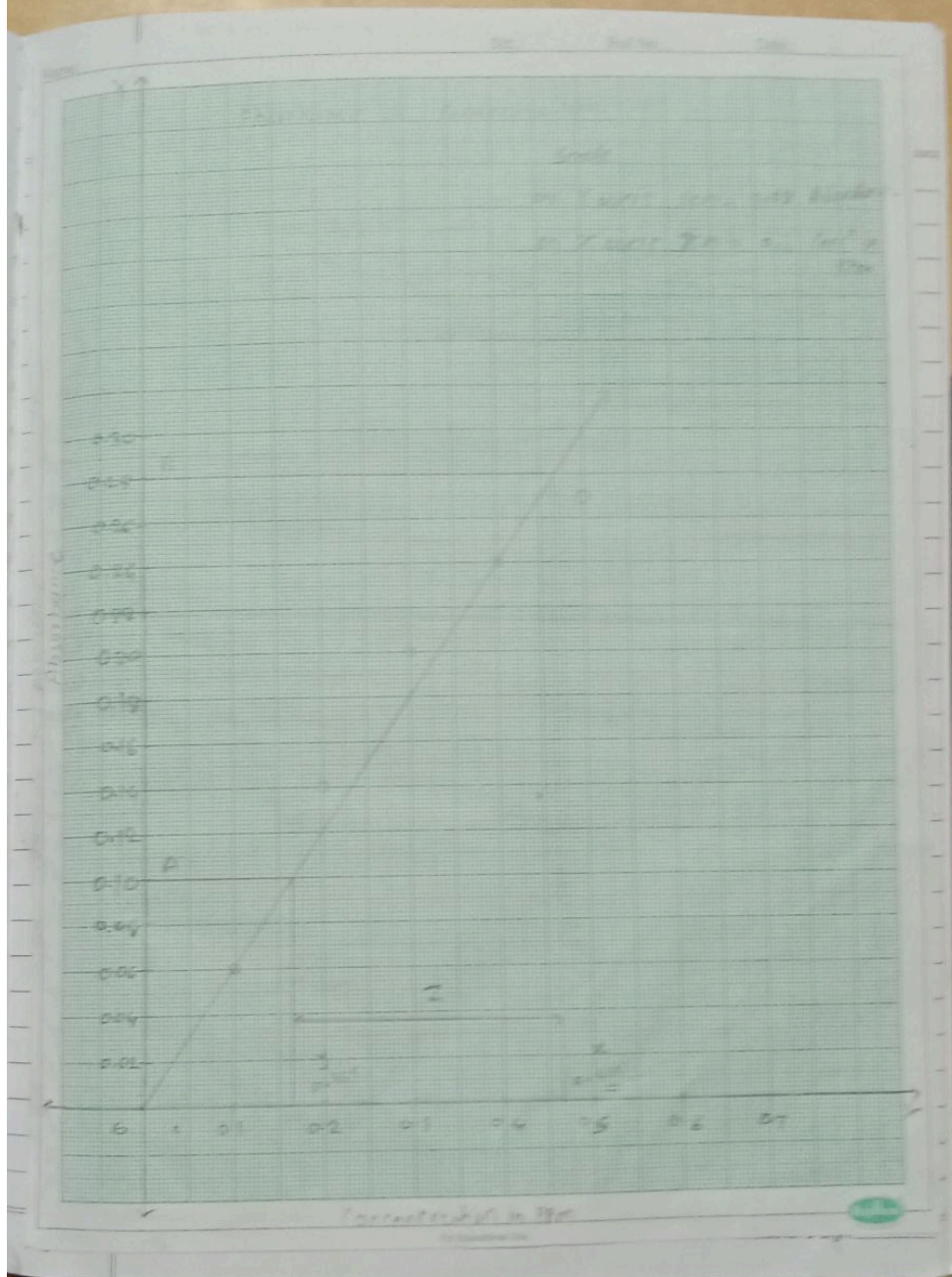
2) Amount of  $Fe(III)$  present in the 10 cm<sup>3</sup> of the diluted sample solution = 0.3 ppm.  
 $\therefore$  Amount of  $Fe(III)$  present in the given sample soln =  $10 \times 0.3 \text{ ppm} = 3 \text{ mg/1000 cm}^3$   
 $\therefore$  100 cm<sup>3</sup> contains 0.3 mg of  $Fe(III)$  ions.

Standard measuring flask No. 7. Add 10 cm<sup>3</sup> 0.05 M K hydrogen phthalate soln. followed by 10 cm<sup>3</sup> of 0.25% 1,10 phenanthroline solution.  
 9) Dilute the soln. to 100 cm<sup>3</sup> with distilled water.  
 10) Measure the absorbance of soln after 10 min. against reagent blank at 515 nm. from the absorbance value, find out amount of  $Fe(II)$  from calibration curve.

b) To determine total  $Fe(II) + Fe(III)$  amount of iron, measuring flask No. 8.

11) Pipette out 10 cm<sup>3</sup> of the dilute sample soln in a 100 cm<sup>3</sup> flask.  
 12) Add 10 cm<sup>3</sup> of hydroxylamine hydrochloride to it & shake.  
 13) Then add 10 cm<sup>3</sup> of 0.05 M potassium hydrogen phthalate and 10 cm<sup>3</sup> of 0.25% 1,10 phenanthroline. reagent to it & shake well.  
 14) Dilute the soln upto the mark with distilled water. and shake well.  
 15) Measure the absorbance at solution at 515 nm. from the absorbance value, find out total amount  $Fe(II)$  and  $Fe(III)$  both, from calibration curve. (x ppm)  
 16) Therefore, amount of  $Fe(III)$  present can be calculated from (x-y) values. (2 ppm).







Results:

1) Amount of  $\text{Fe(II)}$  ion in a given mixture of  $\text{Fe(II)}$  &  $\text{Fe(III)}$   
 $= x = 0.465 \text{ mg}$

2) Amount of  $\text{Fe(III)}$  ion in a given mixture of  $\text{Fe(II)}$   
and  $\text{Fe(III)} = z = 0.108 \text{ mg}$ .  $z = 0.3 \text{ mg}$



### Solution preparation:

① Preparation of 0.01M  $K_2Cr_2O_7$  sol<sup>n</sup>  
 $\text{Molarity} = \frac{\text{wt.}}{\text{M.wt.} \times \text{volume in dm}^3}$

$$0.01 = \frac{\text{wt}}{294 \times 1}$$

$$\text{wt} = 0.01 \times 294$$

$$\therefore \text{wt} = 0.294 \text{ gm.}$$

\* Preparation of sol<sup>n</sup> by using 0.01M  $K_2Cr_2O_7$  solution for  $K_2O$  factor

for 0.001M

$$M_1 V_1 = M_2 V_2$$

$$0.01 \times V_1 = 0.001 \times 100$$

$$V_1 = \frac{0.001 \times 100}{0.01}$$

$$\therefore V_1 = 10 \text{ cm}^3$$

Volume of 0.01M  $KMnO_4$  sol<sup>n</sup> for preparing the following sol<sup>n</sup>

for 0.0005M

$$M_1 V_1 = M_2 V_2$$

$$0.01 \times V_1 = 0.0005 \times 100$$

$$V_1 = \frac{0.0005 \times 100}{0.01}$$

$$\therefore V_1 = 5 \text{ cm}^3$$

for 0.0005M

$$M_1 V_1 = M_2 V_2$$

$$0.01 \times V_1 = 0.0005 \times 100$$

$$V_1 = \frac{0.0005 \times 100}{0.01}$$

$$V_1 = 5 \text{ cm}^3$$

For 0.00025M

$$M_1 V_1 = M_2 V_2$$

$$0.01 \times V_1 = 0.00025 \times 100$$

$$V_1 = \frac{0.00025 \times 100}{0.01}$$

$$V_1 = 2.5 \text{ cm}^3$$

EXPERIMENT No. 4

Spectrophotometry.

Aim:

Simultaneous determination of chromium and manganese in a mixture by spectrophotometrically.

Requirements:

1) 0.005M  $K_2Cr_2O_7$  standard solution:

Prepare 0.1M  $K_2Cr_2O_7$  solution by dissolving 4.94g of  $K_2Cr_2O_7$  in 100 cm<sup>3</sup> standard measuring flask containing 10 cm<sup>3</sup> of 10M  $H_2SO_4$  and dilute to 100 cm<sup>3</sup> with distilled water.

This is the stock  $K_2Cr_2O_7$  solution.

Dilute 5 cm<sup>3</sup> of the stock  $K_2Cr_2O_7$  (0.1M) solution to 100 cm<sup>3</sup> with 1M  $H_2SO_4$  in standard measuring flask. This gives standard 0.005M  $K_2Cr_2O_7$  sol<sup>n</sup> (Cr 105M)

a) 0.005M  $KMnO_4$  sol<sup>n</sup>:

Prepare 0.1M  $KMnO_4$  solution by dissolving 0.315g of  $KMnO_4$  in 100 cm<sup>3</sup> standard measuring flask containing 10 cm<sup>3</sup> of 10M  $H_2SO_4$  and dilute to 100 cm<sup>3</sup> with D.W.

This gives the stock  $KMnO_4$  sol<sup>n</sup>.

Dilute 5 cm<sup>3</sup> of stock  $KMnO_4$  sol<sup>n</sup> (0.1M) to 100 cm<sup>3</sup> with 1M  $H_2SO_4$ . This gives 0.005M standard  $KMnO_4$  sol<sup>n</sup>.

3) 1M Sulphuric acid solution.

a) Sample solution containing chromium (VI) and manganese (VII) in 100 cm<sup>3</sup> standard measuring flask.

5) Six 100 cm<sup>3</sup> standard measuring flasks, burette, 5 cm<sup>3</sup> and 10 cm<sup>3</sup> pipette, spectrophotometer, etc.

Theory:

When there is no interaction between two solutes.

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Observation Table:-

for 0.01M $K_2Cr_2O_7$				
Conc of $K_2Cr_2O_7$ (M)	Volume of $K_2Cr_2O_7$ (ml)	O.D. at 440 nm	$E_{440}^{cm}$	$E_{440}^{cm}$
0.001	10.0	0.28	0.03	280
0.0005	5.0	0.14	0.02	280

for 0.01M $KMnO_4$				
Conc of $KMnO_4$ (M)	Volume of $KMnO_4$ (ml)	O.D. at 525 nm	$E_{525}^{cm}$	$E_{525}^{cm}$
0.0005	5.0	0.04	0.01	80
0.00025	2.5	0.01	0.005	800

Unknown 10 cm <sup>3</sup> of diluted unknown solution	A of mix at 440 nm	A of mix at 525 nm
	0.15	0.04

Calculations:

$$E = \frac{A}{l \times \text{concentration}}$$

\* for  $E_{440} =$

①  $E_{440} = \frac{A}{l} = \frac{0.28}{0.001} = 280 \text{ cm}^2/\text{mol} \cdot \text{dm}^3$

②  $E_{440} = \frac{A}{l} = \frac{0.14}{0.0005} = 280 \text{ cm}^2/\text{mol} \cdot \text{dm}^3$

Average  $E_{440} = \frac{280 + 280}{2} = 280 \text{ cm}^2/\text{mol} \cdot \text{dm}^3$

In a solution, it is possible to determine their conc. in a mixture. The total absorbance of a soln at a given wave length is equal to the sum of absorbance of the individual components present in it. It can be shown as:

$$A_{mix}^{\lambda} = A_1^{\lambda} + A_2^{\lambda}$$

$$A_{mix}^{\lambda} = A_1^{\lambda} \cdot C_1 + A_2^{\lambda} \cdot C_2$$

where  $A_1$  &  $A_2$  refer to the absorbance due to the first and second solute at the particular wavelength. Absorbance is directly proportional to the product of molar absorptivity ( $E$ ) and concentration.  $C$  (moles/dm<sup>3</sup>) Hence for path length of 1cm, we have

$$A_{mix}^{\lambda} = E_1^{\lambda} \cdot C_1 + E_2^{\lambda} \cdot C_2$$

$$A_{mix}^{\lambda} = E_1^{\lambda} \cdot C_1 + E_2^{\lambda} \cdot C_2$$

Where,  $E_1$  and  $E_2$  are molar absorptivities and  $C_1$  &  $C_2$  are concentrations of the two solutes respectively. solution of these simultaneous equations give.

$$C_1 = \frac{(E_2^{\lambda} \cdot A_{mix}^{\lambda}) - (E_2^{\lambda} \cdot A_{mix}^{\lambda})}{(E_1^{\lambda} \cdot A_{mix}^{\lambda}) - (E_2^{\lambda} \cdot A_{mix}^{\lambda})}$$

$$C_2 = \frac{(E_1^{\lambda} \cdot A_{mix}^{\lambda}) - (E_1^{\lambda} \cdot A_{mix}^{\lambda})}{(E_1^{\lambda} \cdot A_{mix}^{\lambda}) - (E_2^{\lambda} \cdot A_{mix}^{\lambda})}$$

The molar absorptivities  $E_1^{\lambda}$ ,  $E_2^{\lambda}$ ,  $E_1^{\lambda}$  and  $E_2^{\lambda}$  can be evaluated from individual standard solutions of two



\* for  $\text{Cr}^{525}$ ;

$$\textcircled{1} \epsilon_{\text{Cr}^{525}} = \frac{A_1}{c} = \frac{0.05}{0.001} = 50 \text{ cm}^2/\text{mol}/\text{dm}^3$$

$$\textcircled{2} \epsilon_{\text{Cr}^{525}} = \frac{A_2}{c} = \frac{0.02}{0.0005} = 40 \text{ cm}^2/\text{mol}/\text{dm}^3$$

$$\text{Average } \epsilon_{\text{Cr}^{525}} = \frac{50+40}{2} = 45 \text{ cm}^2/\text{mol}/\text{dm}^3$$

\* for  $\text{Mn}^{440}$ ,  $\text{Mn}^{440} = \frac{A}{l \times \text{conc.}}$

$$\textcircled{1} \epsilon_{\text{Mn}^{440}} = \frac{A_1}{c} = \frac{0.09}{0.0005} = 180 \text{ cm}^2/\text{mol}/\text{dm}^3$$

$$\textcircled{2} \epsilon_{\text{Mn}^{440}} = \frac{A_2}{c} = \frac{0.01}{0.00025} = 40 \text{ cm}^2/\text{mol}/\text{dm}^3$$

for  $\text{Mn}^{525}$

$$\textcircled{1} \epsilon_{\text{Mn}^{525}} = \frac{A_1}{c} = \frac{0.21}{0.0005} = 420 \text{ cm}^2/\text{mol}/\text{dm}^3$$

$$\textcircled{2} \epsilon_{\text{Mn}^{525}} = \frac{A_2}{c} = \frac{0.20}{0.00025} = 800 \text{ cm}^2/\text{mol}/\text{dm}^3$$

$$\text{Average } \epsilon_{\text{Mn}^{440}} = \frac{180+40}{2} = 110 \text{ cm}^2/\text{mol}/\text{dm}^3$$

$$\text{Average } \epsilon_{\text{Mn}^{525}} = \frac{420+800}{2} = 610 \text{ cm}^2/\text{mol}/\text{dm}^3$$

$$1) \text{Cr} = \frac{A_{\text{mix}}^{440} \cdot \epsilon_{\text{Mn}^{525}} - A_{\text{mix}}^{525} \cdot \epsilon_{\text{Mn}^{440}}}{\epsilon_{\text{Cr}^{440}} \cdot \epsilon_{\text{Mn}^{525}} - \epsilon_{\text{Cr}^{525}} \cdot \epsilon_{\text{Mn}^{440}}}$$

$$= \frac{(0.15 \times 610) - (0.04 \times 110)}{(280 \times 610) - (35 \times 110)}$$

$$= \frac{91.5 - 4.4}{170800 - 3850} = \frac{87.1}{166950}$$

$$\therefore \text{Cr} = 5.28 \times 10^{-4} \text{ mol}/\text{dm}^3$$

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solutes or better from the slopes of their Beer's law plots. The absorbance of the mixture,  $A_{\text{mix}}$  and  $A_{\text{mix}}^{\lambda}$  can be determined experimentally. Hence the concentration of the individual constituents,  $C_1$  &  $C_2$  can be calculated. These relationships are valid only if Beer's law is followed and if the two components behave independently of one another. For the best precision, it is desirable to select the two wavelengths where the ratio of molar absorptivities are maximum. The above principle can be used in the determination of chromium or dichromate ( $\text{Cr}_2\text{O}_7^{2-}$ ) and manganese as permanganate ( $\text{MnO}_4^-$ ) simultaneously, at the wavelengths 440 nm and 525 nm. Beer's law is obeyed in presence of 0.1M sulphuric acid concentration.

Procedure:

I] Determination of molar absorptivity at 440 nm & 525 nm.

1) Take four serially numbered 100 cm<sup>3</sup> standard measuring flask:

2) In flask nos. 1 & 2 add 10 cm<sup>3</sup> and 20 cm<sup>3</sup> of standard potassium dichromate solution ( $5 \times 10^{-3}$  M) and dilute upto the mark with 1M  $\text{H}_2\text{SO}_4$ . This gives 0.0005 M & 0.001 M potassium dichromate solution.

3) In flask Nos. 3 and 4, add 5 cm<sup>3</sup> and 10 cm<sup>3</sup> of standard potassium permanganate sol<sup>n</sup> ( $5 \times 10^{-3}$  M) and dilute upto the mark with 1M  $\text{H}_2\text{SO}_4$  sol<sup>n</sup>. This gives 0.00025 M and 0.0005 M potassium permanganate sol<sup>n</sup>.

Qualin

Teacher's Sign



$$\begin{aligned}
 2) \quad C_{Mn} &= \frac{A_{mix}^{525} \cdot \epsilon_{Cr}^{440} - A_{mix}^{440} \cdot \epsilon_{Cr}^{525}}{\epsilon_{Cr}^{440} \cdot \epsilon_{Mn}^{525} - \epsilon_{Mn}^{440} \cdot \epsilon_{Cr}^{525}} \\
 &= \frac{(0.04 \times 280) - (0.15 \times 35)}{(240 \times 1480) - (128 \times 35)} \\
 &= \frac{11.2 - 5.25}{170800 - 2100} \\
 &= \frac{5.95}{168700} \\
 &= 3.52 \times 10^{-5} \text{ mol/dm}^3
 \end{aligned}$$

for Mn,  
 $1M \text{ KMnO}_4 = 54.94 \text{ gm of Mn}$   
 $\therefore C_{Mn} = 3.52 \times 10^{-5} M \text{ KMnO}_4$   
 $\therefore C_{Mn} = (54.94) \times (3.52 \times 10^{-5})$   
 $= 1.93 \times 10^{-3}$   
 $= 0.00193 \text{ gm of Mn}$   
 $= 1.93 \text{ mg of Mn}$   
 $\therefore \text{Amount of Mn in the given solution} = 1.93 \text{ mg of Mn}$

for Cr,  
 $1M \text{ K}_2\text{Cr}_2\text{O}_7 = 2 \times \text{wt of Cr}$   
 $= 2 \times 52$   
 $= 104 \text{ gm of Cr.}$   
 $\therefore C_{Cr} = (5.28 \times 10^{-4}) M \text{ K}_2\text{Cr}_2\text{O}_7$   
 $= 0.05702 \text{ gm of Cr}$   
 $= 57.02 \text{ mg of Cr.}$

$\therefore \text{The amount of Cr in the given sol}^n = 57.02 \text{ mg.}$

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- 4) Measure the absorbance (O.D.) of each sol<sup>n</sup> separately at both 440 nm and 525 nm using 1M H<sub>2</sub>SO<sub>4</sub> as blank.
- 5) Using the absorbance values, calculate the molar absorptivity in each case and determine the mean value for dichromate and permanganate ions at the two wavelengths.

II] Determination of concentration of chromium (VI) and manganese (VII) in the mixture.

- 6) Dilute the given sample sol<sup>n</sup> upto the mark with 1M H<sub>2</sub>SO<sub>4</sub> in 100 cm<sup>3</sup> standard measuring flask.
- 7) Transfer 10 cm<sup>3</sup> of this diluted sol<sup>n</sup> to another 100 cm<sup>3</sup> standard measuring flask numbered as 5 and dilute upto the mark with 1M H<sub>2</sub>SO<sub>4</sub>.
- 8) Measure the absorbance of this sol<sup>n</sup> at 440 nm & 525 nm using 1M H<sub>2</sub>SO<sub>4</sub> as blank.
- 9) Using the value of absorbance at the two wavelengths and molar absorptivities of chromate & permanganate ions at the corresponding wavelengths, calculate the concentration of these ions in the given sample.

Result:

① Amount of Cr(VI) present in the given sol<sup>n</sup>.  
 $= 57.02 \text{ mg}$

② Amount of Mn(VII) present in the given sol<sup>n</sup>.  
 $= 1.93 \text{ mg}$

Teacher's Sign.

Galathea